

Parametric study on the mixed solvent synthesis of ZIF-8 nano- and micro-particles for CO adsorption: A response surface study

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Electronic Supplementary Material

Table S1. Structural parameters of ZIF-8 synthesized in methanol by using N₂ adsorption isotherm and BJH method

| | V_p / cm ³ /gr | a_p / m ² /gr | $r_{p,peak}$ (Area) / nm |
|-------|--------------------------------|--------------------------------|-----------------------------|
| meso | 0.057615 | 12.802 | 1.21 |
| micro | 0.5107 | $a_1 = 1657$ $a_2 = 8.5258$ | 0.30 |

Table S2. Hansen solubility parameters of applied solvents (methanol and water) along with 2-MeIM ligand

| Component | Hansen Solubility Parameters (HSPs) / MPa ^{0.5} | | | Ref. |
|-----------|--|------------|------------|------|
| | δ_D | δ_P | δ_H | |

| | | | | |
|----------|------|------|------|-----|
| Methanol | 15.1 | 12.3 | 22.3 | [1] |
| Water | 15.1 | 20.4 | 16.5 | [1] |
| 2-MeIM | 18.8 | 10.7 | 9.7 | [2] |

Table S3. Hansen solubility parameters of methanol- water mixtures with different amount of water, and solubility parameter difference (Ra) between the mixtures and 2-MeIM

| Solvent | Hansen Solubility Parameters (HSPs) / MPa ^{0.5} | | | R _a |
|-----------------------------|--|------------|------------|----------------|
| | δ_D | δ_P | δ_H | |
| Pure Methanol | 15.1 | 12.3 | 22.3 | 14.7 |
| Methanol-Water (25% vol) | 15.1 | 14.3 | 20.8 | 13.86 |
| Methanol-Water (50% vol) | 15.1 | 16.3 | 19.5 | 13.51 |

Table S4. Analysis of variance (ANOVA) of response surface reduced cubic model for predicting the yield of ZIF-8 synthesis

| Source | Sum of squares | df | Mean square | F value | p-value |
|---------------------------|----------------|----|-------------|---------|---------|
| Model | 7658.18 | 12 | 638.18 | 3429.24 | 0.0003 |
| A: 2-MeIM content | 1010.60 | 1 | 1010.60 | 5430.44 | 0.0002 |
| B: Sodium formate content | 30.58 | 1 | 30.58 | 164.33 | 0.0060 |
| C: Water content | 37.70 | 1 | 37.70 | 202.58 | 0.0049 |
| AB | 0.27 | 1 | 0.27 | 1.45 | 0.3513 |

| | | | | | |
|------------------|---------|----|---------|----------|----------|
| AC | 1025.92 | 1 | 1025.92 | 5512.74 | 0.0002 |
| BC | 85.01 | 1 | 85.01 | 456.79 | 0.0022 |
| A ² | 1124.96 | 1 | 1124.96 | 6044.93 | 0.0002 |
| B ² | 814.24 | 1 | 814.24 | 4375.27 | 0.0002 |
| C ² | 2601.74 | 1 | 2601.74 | 13980.32 | < 0.0001 |
| A ² B | 27.83 | 1 | 27.83 | 149.52 | 0.0066 |
| A ² C | 190.13 | 1 | 190.13 | 1021.63 | 0.0010 |
| AB ² | 323.34 | 1 | 323.34 | 1737.47 | 0.0006 |
| Pure Error | 0.37 | 2 | 0.19 | | |
| Cor total | 7658.55 | 14 | | | |

Table S5. Analysis of variance (ANOVA) of response surface reduced cubic model for predicting the particle size of ZIF-8

| Source | Sum of squares | df | Mean square | F value | p-value |
|---------------------------|----------------|----|-------------|---------|---------|
| Model | 1.838E+007 | 12 | 1.531E+006 | 1752.00 | 0.0006 |
| A: 2-MeIM content | 8.556E+006 | 1 | 8.556E+006 | 9789.00 | 0.0001 |
| B: Sodium formate content | 58709.29 | 1 | 58709.29 | 67.17 | 0.0146 |
| C: Water content | 19937.44 | 1 | 19937.44 | 22.81 | 0.0412 |
| AB | 443.52 | 1 | 443.52 | 0.51 | 0.5501 |

| | | | | | |
|------------------|------------|----|------------|---------|--------|
| AC | 13665.61 | 1 | 13665.61 | 15.63 | 0.0584 |
| BC | 61504.00 | 1 | 61504.00 | 70.37 | 0.0139 |
| A ² | 3.693E+006 | 1 | 3.693E+006 | 4224.95 | 0.0002 |
| B ² | 2.032E+006 | 1 | 2.032E+006 | 2324.64 | 0.0004 |
| C ² | 2.718E+006 | 1 | 2.718E+006 | 3109.83 | 0.0003 |
| A ² B | 29001.95 | 1 | 29001.95 | 33.18 | 0.0288 |
| A ² C | 3.607E+005 | 1 | 3.607E+005 | 412.62 | 0.0024 |
| AB ² | 2.722E+006 | 1 | 2.722E+006 | 3114.46 | 0.0003 |
| Pure Error | 1748.13 | 2 | 874.06 | | |
| Cor total | 1.838E+007 | 14 | | | |

Fig S1. FE-SEM images of ZIF-8 particles synthesized in different conditions of experimental design along with particle size distribution

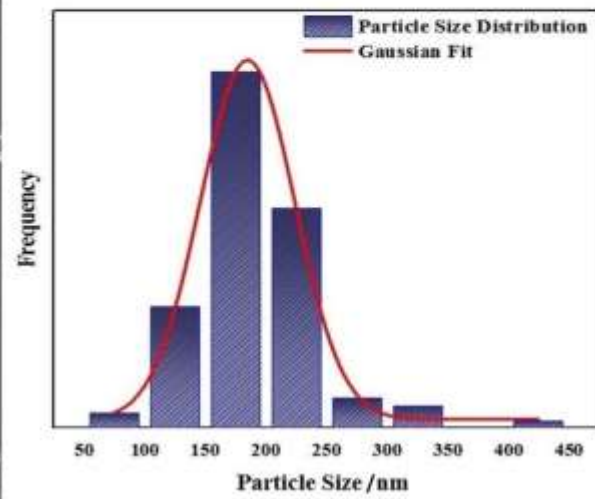
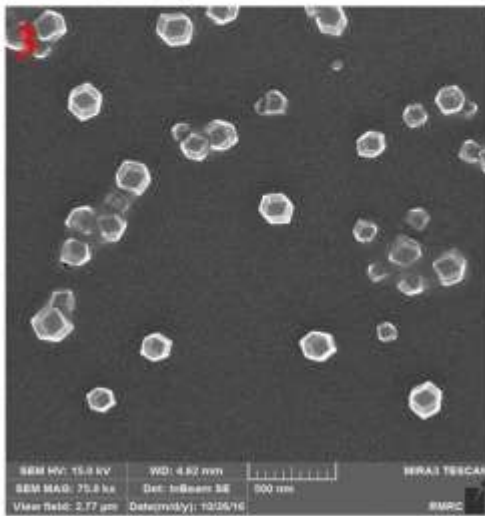
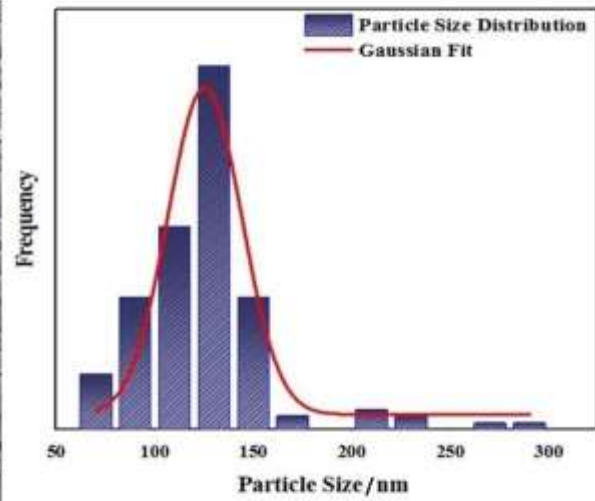
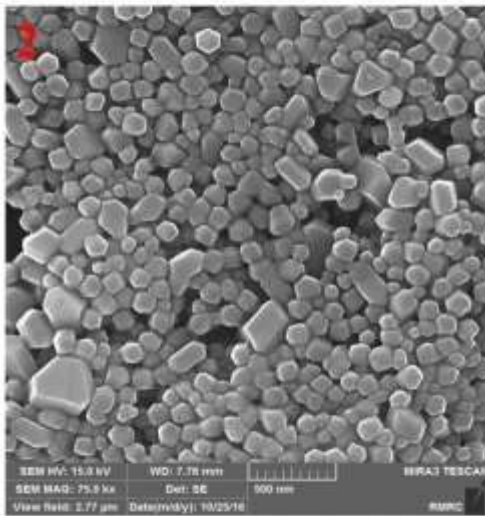
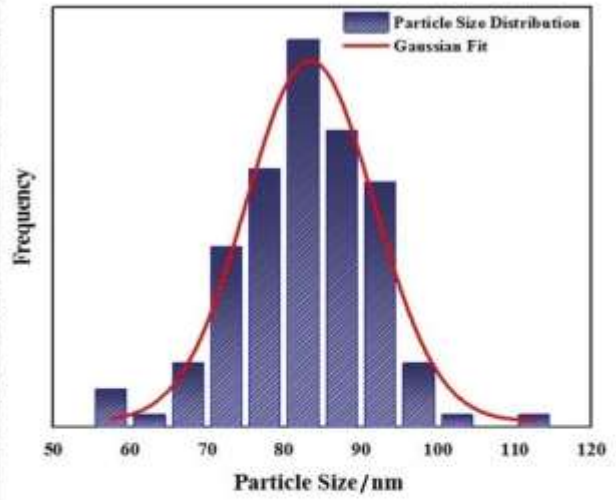
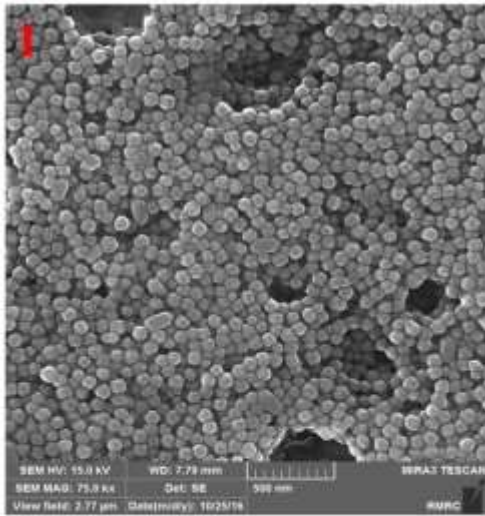


Fig S1. Continued

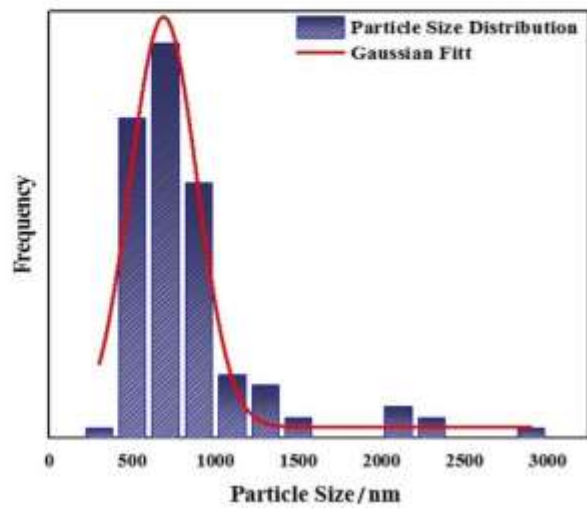
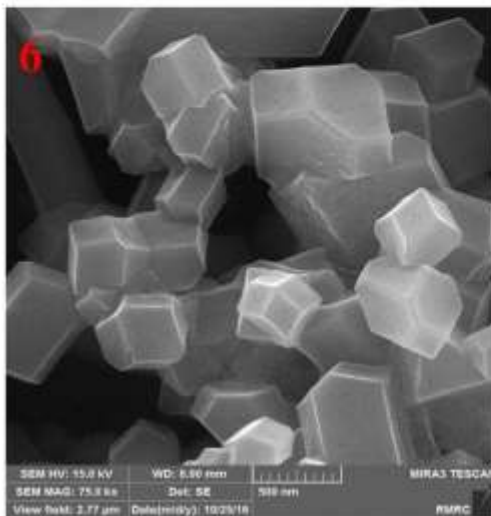
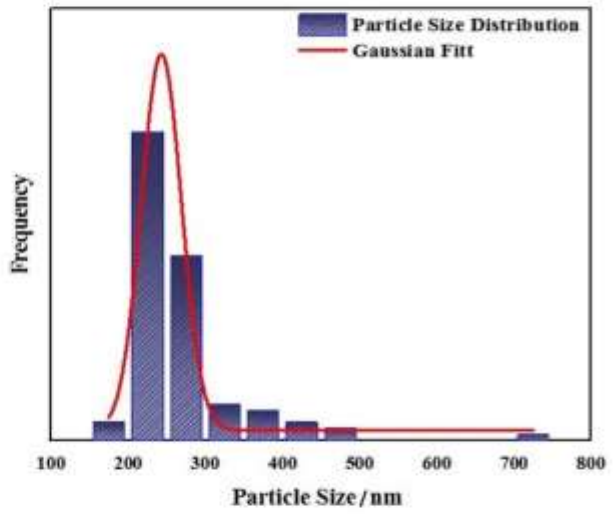
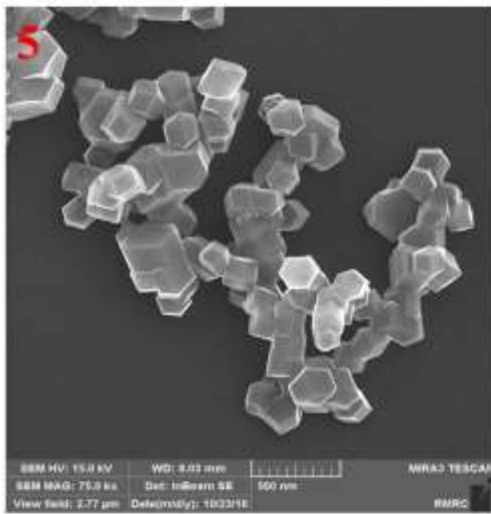
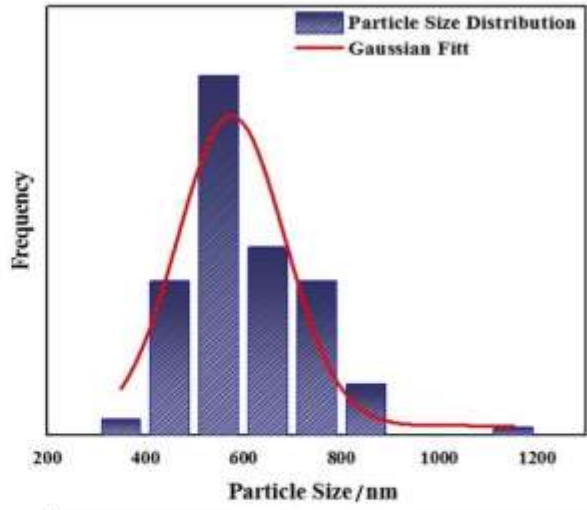
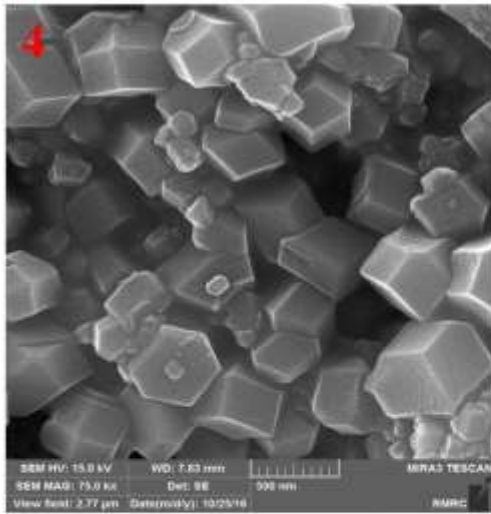


Fig S1. Continued

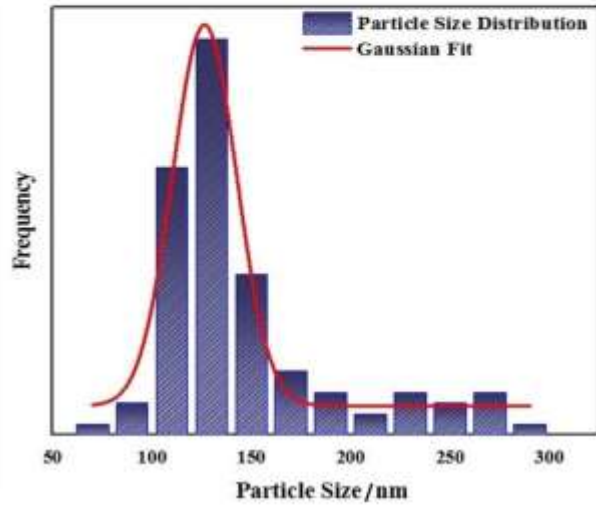
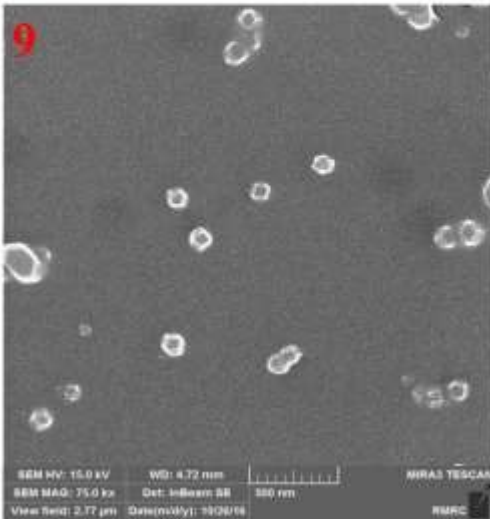
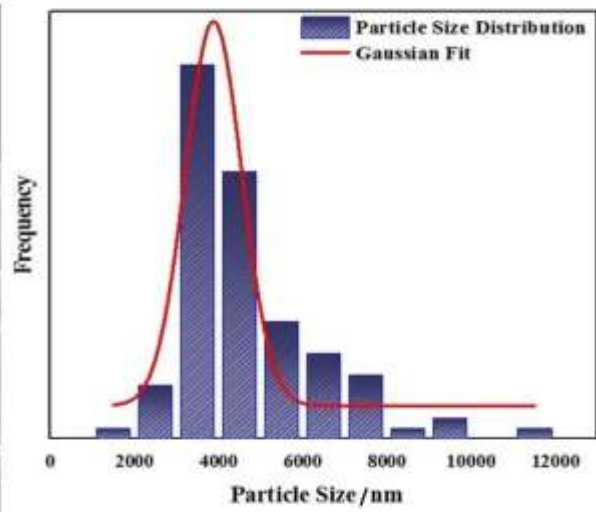
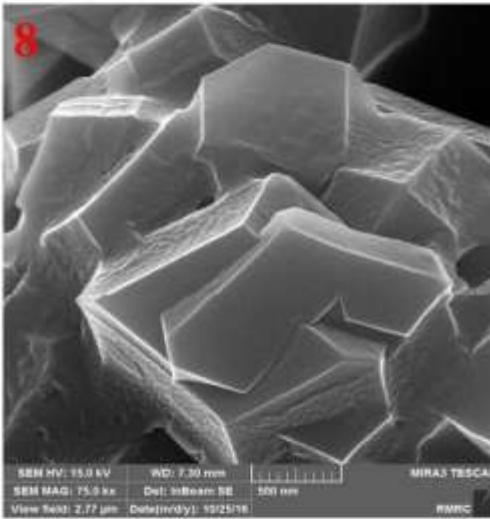
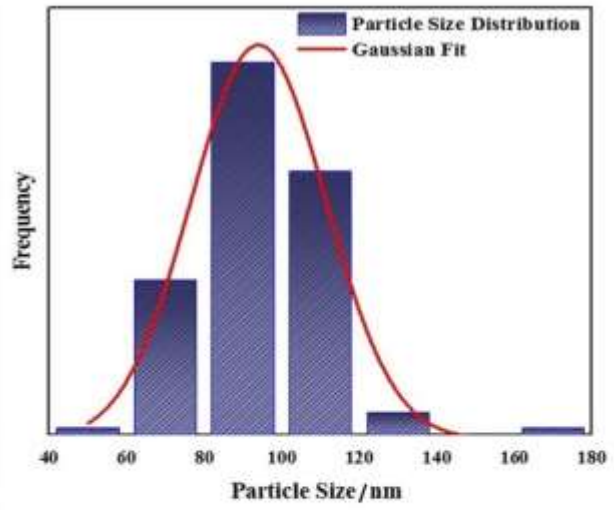
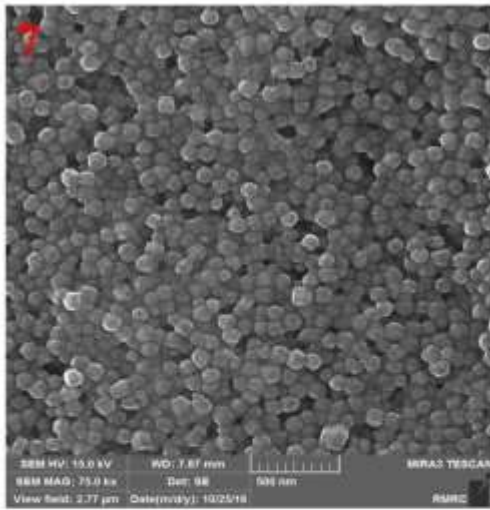


Fig S1. Continued

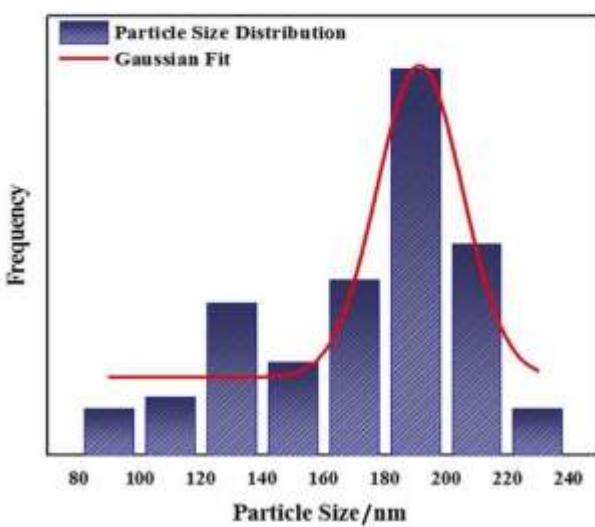
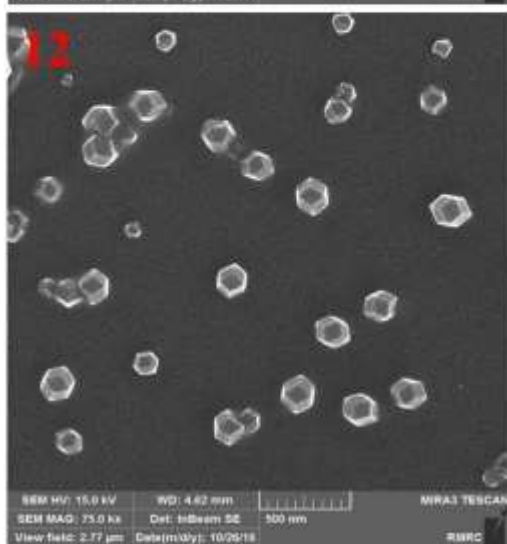
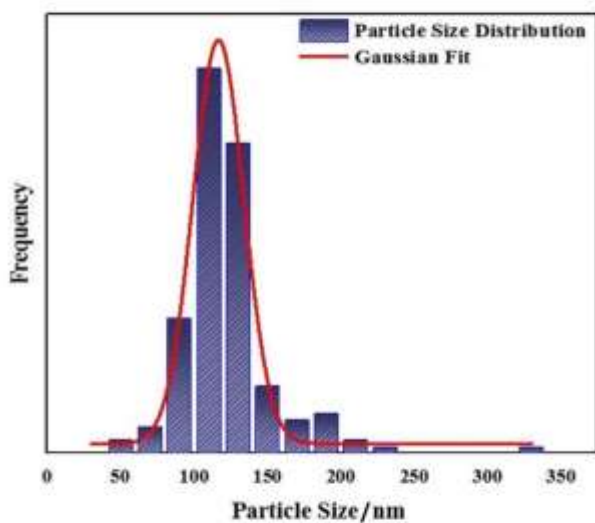
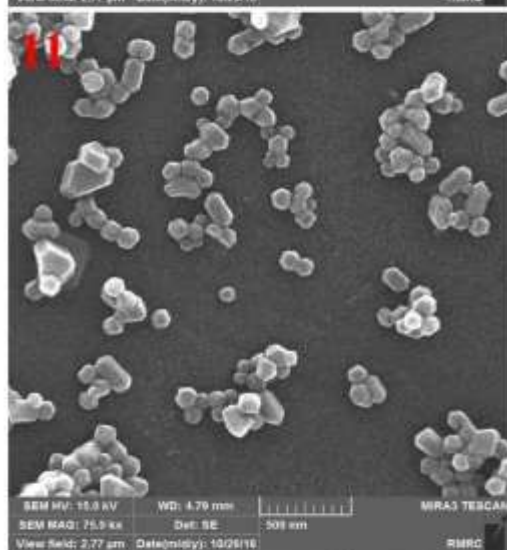
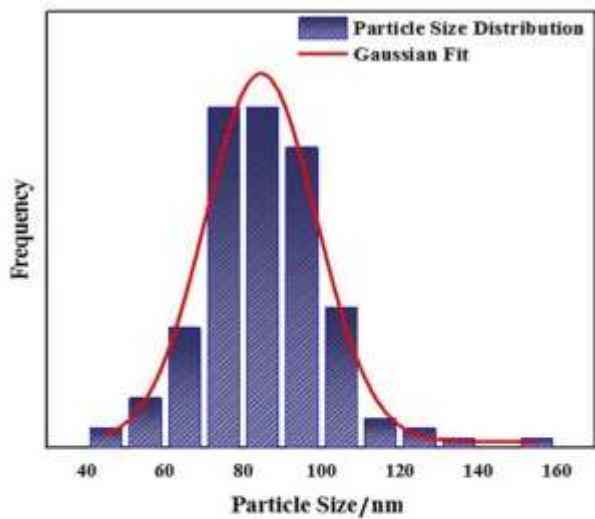
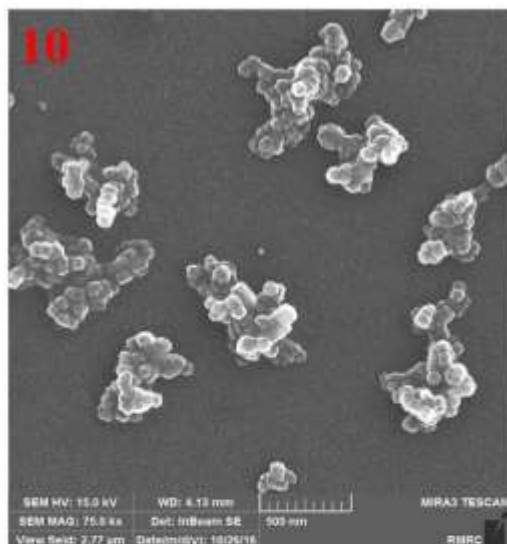


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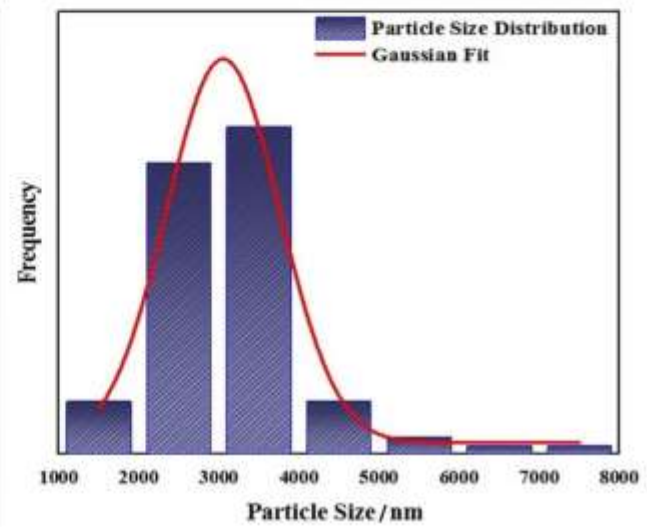
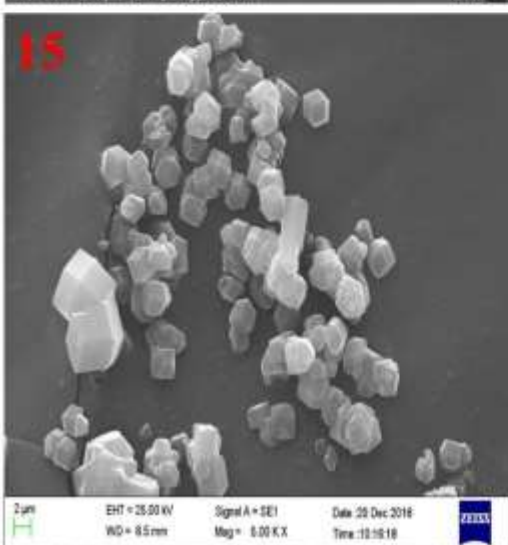
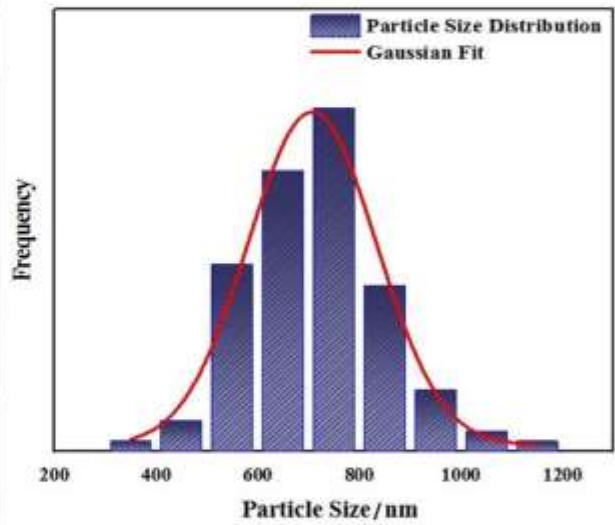
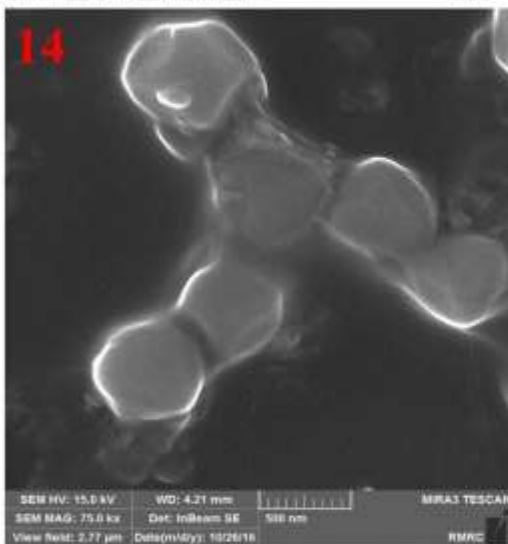
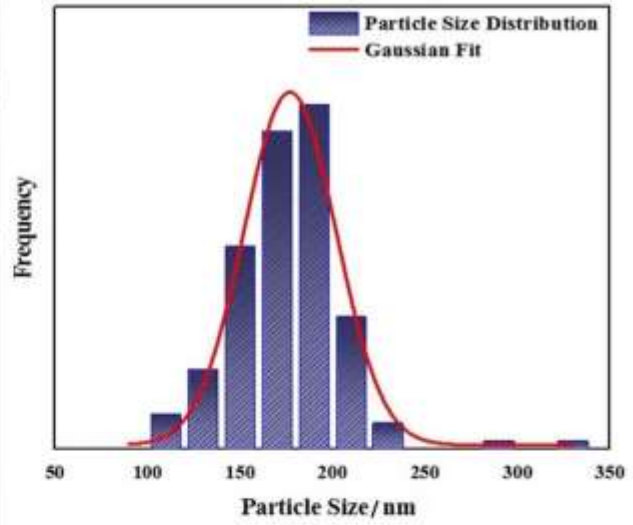
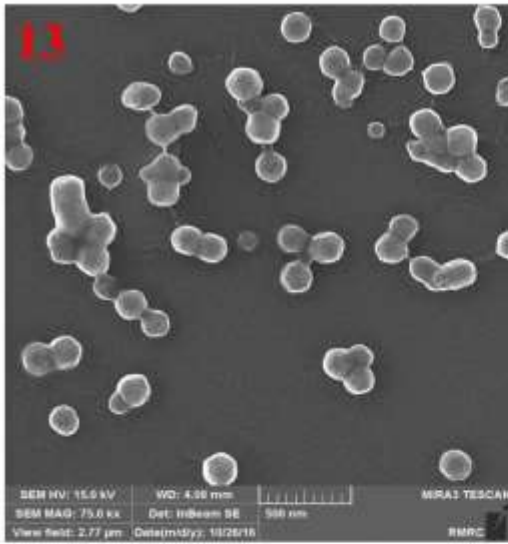


Fig S1. Continued

Table S6. Synthesis conditions of ZIF-8 particles with different particle size for CO adsorption

| Adsorbent (mean particle size) | Solvent / % vol | | Reagents/ zinc nitrate molar ratio | |
|-----------------------------------|-----------------|-------|------------------------------------|----------------|
| | Methanol | Water | 2-MeIM | Sodium formate |
| ~100 nm | 100 | 0 | 6.23 | 0 |
| ~500 nm | 100 | 0 | 5.5 | 4 |
| ~1000 nm | 75 | 25 | 2 | 3.8 |

References:

1. Hansen C M. Hansen Solubility Parameters: a user's handbook. Boca Raton: CRC Press, 2007.
2. Paseta L, Potier G, Abbott S, Coronas J. Using Hansen solubility parameters to study the encapsulation of caffeine in MOFs. *Organic & Biomolecular Chemistry*, 2015, 13(6): 1724-1731